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Accelerated Precipitation in Fatigue
of Precipitation Hardening Alloy
Aluminum 4 Per Cent Copper

By S. R. Valluri

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Final Report - June 1960

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ACCELERATED PRECIPITATION IN FATIGUE
OF PRECIPITATION HARDENING ALLOY
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SUMMARY

Previously experimental evidence has been reported which shows that under conditions of fatigue, precipitation hardening alloys which are essentially in a metastable condition suffer substantial precipitation in certain regions. This process leaves some regions devoid of the precipitation hardening elements and it has been conjectured on the basis of metallographic evidence that fatigue cracks are nucleated in these regions depleted of the solute atoms of the hardening elements. Attempts have been made to follow this precipitation process in a quantitative manner using internal friction techniques. Experimental evidence is presented to show that the precipitation does in fact take place. The results are preliminary and the indications so far are that if a solution treated and quenched test specimen is subjected to fatigue, precipitation takes place at a faster rate than may be expected under simple room temperature ageing and this effect is also dependent upon stress. Specifically, it appears that if the stress amplitude is high, precipitation takes place at a faster rate. More confirmatory work is needed to explore the idea thoroughly.

INTRODUCTION

In 1956, in a symposium held in Columbia University on Fatigue in Aircraft Structures, two significant papers were presented. One was that of Forsyth (Ref. 1) on the mechanism of fatigue in aluminum and aluminum alloys and the other was that of Hanstock on the effects preceding fatigue failure of high strength aluminum alloys. On the basis of experimental investigations Forsyth conjectures that "fatigue stressing not only moves the dislocations but also causes a migration of solute atoms from the heavily deformed regions of the material". This is a catastrophic process in the sense that the depletion of the matrix in turn leads to heavy deformation and this depletion will result in cracking which may be accompanied by a marked extrusion effect. Presumably the fatigue cracks occur in the softened zones produced by the depletion of solute atoms. Hanstock (Ref. 2), who made measurements of damping at engineering stress levels on precipitation hardening alloys, showed on the basis of comparison between the variation of damping of the test specimens under fatigue conditions and the variation of damping under conditions of ageing at elevated temperatures, the material behavior was substantially the same. He argues on this basis that a precipitation hardening alloy subjected to fatigue behaves as if it were subjected to elevated temperatures. Since the effect of elevated temperatures on these alloys which are in a metastable condition is one of enhanced precipitation, he argues that fatigue stress also must give rise to such enhanced precipitation. This enhanced precipitation will obviously leave certain regions devoid of the precipitation hardening elements and the soft spots so

generated may be considered as potential damage nuclei in the sense that the fatigue cracks may be expected to start in these regions. In fact Hanstock has been able to show by careful sectioning that this is indeed the case. He presents pictures in which there are cracks near the surface and close to them, precipitation bands.

If the accelerated precipitation is responsible for the degeneration of the precipitation hardening alloys, the central idea that appears worth investigating is, what is the rate at which this precipitation takes place? If it is possible to ascertain this rate under conditions of fatigue, we may conceivably be in a position to determine the rate at which "soft spots" are generated in these materials. This becomes pertinent since when once the "soft spots" occur, the material generally forgets that it is subjected to fatigue stresses everywhere, and the damage gets concentrated in these "soft spots" and the process becomes catastrophic. In several of these "soft spots" microscopic fatigue cracks probably are generated fairly early in the life of the specimen and a stage comes where these microscopic cracks join together much like the tributaries of a river leading to a catastrophic failure.

In order that we may pursue the idea in a sensible manner we need to understand two additional problems. They are respectively the process of precipitation hardening and the principles and techniques of internal friction.

Since the discussion of precipitation in a general manner is rather complex and since we are interested specifically in aluminum alloys we will restrict our attention to the binary alloy of aluminum and 4 per cent copper. The structural changes during ageing of this alloy

have been extensively investigated and the following is a very brief review of the problem. Somewhat more extensive information is given by Berry and Nowick (Ref. 3) and a fairly detailed review of the problem is presented by Hardy and Heal (Ref. 4).

The solubility of copper in aluminum decreases progressively from about 5.65 per cent at 548°C to less than 0.25 per cent at normal room temperature. The phase in equilibrium with the aluminum rich solid solution is the intermetallic compound CuAl_2 familiarly called θ phase. However when an aluminum alloy containing 4 weight per cent copper is aged at temperatures below 300°C simple precipitation of the θ phase does not occur. Instead one or more of three other structures may be formed depending upon the ageing temperature.

G. P. I Zones: Room temperature ageing from a quenched condition from an elevated temperature where all the copper is taken into solution was found by Guinier and Preston in 1938 to give rise after about 5 hours to local enrichment in copper of small areas distributed on the $\{100\}$ planes of the matrix. This process was found to reach a constant condition in a few days after which no further change was detected. The thickness of the zones was estimated to be about 8A and the diameter after room temperature ageing to be about 50A. Preston concludes on the basis of available information that the fully formed G. P. I zones are about 200A apart in the matrix.

It is convenient at this state to discuss a phenomenon called "reversion" which is of considerable importance in the work. It has been found for example that if hardness measurements are made on Al 4 per cent Cu immediately after quenching, the hardness continues

to rise gradually and reaches a constant value in a few days. If such an alloy after reaching a constant condition is heated to an elevated temperature of about 200°C for a few minutes, the hardness is found to go down very quickly to a low value before it starts rising again. The accompanying structural changes are found from investigations to be the disappearance of the G. P. I zones. If the copper in the alloy immediately after quenching is termed free copper, as a matter of convenience, the phenomenon of reversion may be considered to be the reversion of the copper in G. P. I zones to one of free copper. Reversion may be expected when the temperature is raised either to a value above which the particles that have formed become of subcritical size for stability or above which the structure is no longer even metastable.

G. P. II Zones: This structure was first discovered by Guinier in specimens which had been aged at room temperature and then heated for several days at 100°C or for some hours at 150°C . He suggested that the G. P. II zones consist of copper enriched regions where ordering has occurred, in the sense that copper enriched planes alternated with impoverished planes in a regular or orderly sequence, the repetition occurring after every fourth plane. The detailed structure and the lattice spacing is of no special interest here.

The θ' Phase: It was concluded by Preston that the structure of this phase is slightly tetragonal but otherwise of the calcium fluoride type (Ca F_2) containing aluminum and copper in the proportion 2 to 1. The precipitation of θ' phase in the form of platelets on the $\{100\}$ matrix planes is apparently shown in the photographs of Calvet et al (according to Berry and Nowick, Ref. 3).

The general sequence of the structures during ageing has been established as G. P. I, G. P. II, θ' and θ . No more than two neighbors are known to co-exist and as the temperature of the ageing is increased gradually fewer of the structures are detected. Below roughly 165°C , G. P. I is the first structure detected; between 165°C and 220°C it is G. P. II; and above 220°C it is the θ' phase. The temperature above which the θ phase precipitates directly is probably in the range of 350°C to 400°C . It was also found that brief excursions can be made above 165°C a few times without causing the formation of G. P. II zones. This is of vital importance in this work since such excursions are necessary in order to determine the internal friction and one must make sure that the measurements themselves do not introduce any changes at lower temperatures. Where the G. P. I is formed first, nucleation of G. P. II from G. P. I appears to be probable.

With regard to the possibility of the transformation of G. P. II to θ' Silcock, Heal and Hardy have shown that θ' platelets, when formed subsequently to G. P. II, have an initial thickness, greater than that of G. P. II, a fact suggesting that θ' can be formed by transformation of G. P. II. Guinier has produced convincing evidence that θ' phase transforms allotropically to the θ phase. The details of these various transformations have been well summarized by Berry and Nowick (Ref. 3).

is known as Zener relaxation. Zener's original hypothesis that this is due to the stress induced reorientation of nearest pairs of neighboring solute atoms faces some difficulties. The theory is not well developed for this phenomenon but the experiments point to the following facts among others.

There is no clear cut method for predicting which particular solid solution should show the Zener relaxation. Thus for example while aluminum copper system and aluminum magnesium system show the phenomenon, an aluminum zinc system does not show it.

The relaxation strength is strongly dependent on the composition of the alloy. It has been shown in several instances that this is proportional to the square of the concentration of the solute. Because of the concentration dependence this phenomenon exhibits itself rather well in solutions containing in excess of 10 atomic per cent solute.

The absolute temperature of the Zener relaxation peak is directly proportional to the activation energy of the relaxation at a testing vibrational frequency of 1 c. p. s.

Berry and Nowick (Ref. 3) conducted extensive investigations in an effort to relate the variation of internal friction under different conditions of ageing of the precipitation hardening solid solution aluminum 4 per cent copper. It is not considered relevant to review their publication here but is necessary to point out some of their results as they have a bearing on the work to be reported.

The alloy aluminum 4 per cent copper, which is a substitutional solid solution, is susceptible to stress induced ordering. Accordingly the internal friction peak measurements of such an alloy in a solution

treated and quenched condition exhibit the characteristics of Zener relaxation. For single crystal specimens the height of the peak is strongly sensitive to the directions of the crystallographic axes with respect to the specimen axis. On ageing the height of the peak decreases as the square of the concentration of copper that remains freely dispersed (free copper) in solid solution. The height of this initial peak is particularly suited to the study of the decrease of the super saturation of the matrix in the early stages of ageing. Since the process of reversion converts the G. P. I zones to free copper, the decrease in the height of the peak is directly proportional to the amount of G. P. II zones formed in the matrix. There is evidence for quenched-in lattice defects (possibly single vacancies) which greatly decrease the mean atomic jump time. The defects decay out rapidly on heating the quenched specimen and the equilibrium concentration is obtained in less than 5 minutes at 200°C. This phenomenon manifests itself as "initial instability" and does not particularly concern us here if the internal friction is measured during the decreasing part of the temperature cycle from an elevated temperature. As the precipitation progresses one may observe the growth of a second broader peak and a rise in the background internal friction. These may be associated with the precipitation of the θ' phase. The height of the second peak is at least roughly proportional to the amount of θ' precipitation. The height of the second peak is found to be reduced by the transformation of θ' phase to θ phase and the peak is not found in specimens containing θ phase alone.

With this as background we may now procede to discuss the fatigue of solution treated and quenched aluminum 4 per cent copper.

In the quenched condition it is reasonable to assume that the copper is fairly well dispersed through the test specimen which is polycrystalline. The specimen may be expected to contain point defects such as lattice vacancies which would anneal out on first heating. The specimen will also contain dislocations. There will be some period at room temperature where the specimen, while being prepared for measurements of internal friction, will suffer ageing and therefore some of the free copper may be expected to transform to G. P. I zones. If the time at room temperature is not too large, as has been stated already, the free copper that transforms to G. P. I may be considered to be negligible. After a prolonged period of ageing at room temperature substantial amounts of free copper may be expected to be transformed to G. P. I. If the internal friction of such a specimen is determined by first raising the temperature to 200°C in about 10 minutes and the specimen kept at that temperature 5 minutes all the point-defects anneal out and the G. P. I zone copper reverts back to free copper by virtue of the property of reversion of G. P. I to free copper whenever the temperature is raised to 200°C for a few minutes. Thus when the internal friction peak is determined, the height of the peak should represent fairly accurately the amount of free copper that exists in the solid solution. That is, if we started with an aluminum 4 weight per cent copper and solution treated it carefully so that all the copper is taken into solution prior to measurement, the peak height should then represent the amount of free copper. Thus if the height of the peak is δ then $\delta \propto C^2 = .04^2$. It appears that this measurement can be repeated about 4 to 5 times without substantially changing the height of the peak. In the process of determining the height of the peak 4 or 5

times, the specimen is held at temperatures above 165°C for about a total of 2 hours. Experimental observations indicate, as Berry and Nowick pointed out in their tests and as was found in this work also, that these periodic brief excursions above the temperature of 165°C do not give rise to the formation of G. P. II zones. This conclusion was drawn on the basis that the variations when the height of the peak was determined 4 times (with one day intervals) were only about 5 per cent as shown in Fig. 1. In other words, the initial peak is fairly stable and it can be used to follow the precipitation processes due to thermal effects or otherwise.

If, after determining the height of the peak once, instead of ageing the material at room temperature the material is subjected to fatigue, as observed by Forsyth (Ref. 1), precipitation at an accelerated rate may be expected to occur. At this stage it is necessary to discuss an effect which may conceivably have a bearing on the discussion. This is the influence of the dislocations on the mode of precipitation. For example it has been known for some time that in interstitial solid solutions of the type α -iron and carbon the height of the internal friction peak for stress induced ordering (Ref. 7) can be used for study of the degree of precipitation of the excess solute from the solvent on ageing; in this case the precipitation is random. On the other hand, if the material is subjected to a certain amount of prior cold work, say of the order of 5 per cent, the precipitation does not occur in the manner indicated. Cottrell and Bilby (Ref. 7) suggest on the basis of theoretical studies and Harper (Ref. 7) experimentally confirmed that the precipitation of solute atoms preferentially to edge

dislocations takes place giving rise to atmospheres and anchoring the dislocations. This process also incidentally gives rise to the characteristic properties of yield drop and strain ageing. Therefore if we argue that preferential precipitation along dislocations must necessarily give rise to yield drop and strain ageing, it seems plausible also to argue conversely that if a particular material does not show yield drop and strain ageing, preferential precipitation along edge dislocations will not take place. The consequence of this argument is that in substitutional solid solutions of the face centered cubic metals which do not generally show yield drop and strain ageing, preferential precipitation along the edge dislocations may not take place.* This amounts to saying that in aluminum 4 per cent copper when subjected to fatigue stressing at moderate stress levels, preferential precipitation along edge dislocations may be neglected. The precipitation may then be expected to proceed at an accelerated rate along the traditional path namely free copper \rightarrow G. P. I \rightarrow G. P. II \rightarrow θ' \rightarrow θ . However it must be stressed that the fatigue process as prior reports indicated (Ref. 8) may be expected to give rise to an increase in the internal friction at room temperature. A substantial part of this damping may be expected to

* It appears from a personal communication from Mr. A. J. Nock of ALCOA Research Laboratories that breaks in the stress-strain curves do occur if the tensile test is performed within one or two hours after quenching and that it does not occur afterwards. In the present series of tests the first measurement of δ vs T was performed within one to three hours and the specimen was subjected to its first period of fatigue stressing after about a minimum period of 5 hours. Hence it does not seem unreasonable to assume that the migration of solute atoms to the edge dislocations (a process that is characteristic of body centered cubic interstitial alloy structures) may not occur in aluminum 4 per cent copper which is a typical face centered cubic alloy of the substitutional type.

decrease due to the recovery process (Ref. 9). However this recovery can be accelerated by a short raise in the temperature of the test specimen. Care must be taken to see that this does not affect the precipitation itself. Since prior work showed that several hours of ageing at 150°C is needed to form G. P. II zones, a brief hold at this temperature or less may be expected to enhance the recovery but leave the precipitation process unchanged due to this treatment.

Under these conditions the decrease in the height of the internal friction peak associated with stress induced ordering may be used for the study of the precipitation of copper in aluminum 4 per cent copper under fatigue. Specifically, whatever precipitation process is observed due to thermal effects may also be observed due to mechanical effects provided these mechanical effects are of such a nature that they can enhance diffusion processes in the alloy. The decrease of the initial peak under fatigue conditions over any normally expected decrease due to rest at the fatigue test temperature may be associated with the formation of the G. P. II zones. Any formation of a second broader peak at a lower temperature may correspondingly be associated with the formation of θ' phase.

The above discussion is of some importance since it appears that this method may be used to follow the basic mechanism of fatigue in a quantitative manner. So far whenever a quantitative value was desired, almost invariably one took recourse to stress vs number of cycles to failure as a means for describing the fatigue phenomenon. We know now that fatigue is a highly inhomogeneous process and a measurement like cycles to failure will give but a crude insight into the problem. On the other hand, if the internal friction technique can be successfully used,

since the accelerated precipitation is due to the fatigue process only, we are in a position to make macroscopic measurements that directly relate to microscopic phenomena and hence get a better insight into the basic mechanism of fatigue.

How successfully the above principle can be used is dependent upon experimental techniques. Normally, in the studies of internal friction it is customary to use wires of about 0.01 inch in diameter and it is relatively easy to reduce the background damping to a substantially low value and keep it so. However, in fatigue the specimen must have reasonable diameter so that stresses of normal engineering magnitude can be imposed without undue elastic deformations. This would in effect mean a compromise specimen size between that for typical internal friction measurements and fatigue measurements. This gives rise to numerous problems in the grips which are a source of enhanced background damping. Everytime the specimen is heated and cooled in the process of measurement, some of the screws holding the test specimen in the testing machine become loose and gives rise to a change of background damping. Also the fatigue stresses introduce an increase in damping. In the initial stages when this is only due to the effect of dislocations and not incipient cracks, it is not a serious problem due to the recovery effect which can be enhanced by a short hold at an elevated temperature. Nevertheless, the aspect of the background damping, because of the quantitative nature of the measurements, remains a vexing problem.

It may be stressed in passing, that strictly speaking it is not possible to compare the results between various specimens except on a percentage basis. The variations in damping of polycrystalline test

specimens is so large that it is almost virtually impossible to obtain two specimens with substantially similar initial internal friction curves.

EXPERIMENTAL EQUIPMENT

The experimental equipment was described in some detail in earlier reports (Refs. 8 and 9). Because of the nature of the testing, some changes were made. It will be clear from the discussion in the introduction section that the time at any specific temperature is of considerable importance since this may conceivably affect the test specimen. A reasonably uniform temperature has to be maintained through the length of the specimen. Because of the openings in the furnace at the ends, a flow of air is created within the furnace causing a gradient. Since a temperature variation of more than 1°C is not tolerable, this gradient problem has been solved by creating an opposite gradient of hot air in the furnace. To control the temperature effectively, five separate heating elements are used with a blower fan to create an airflow in the suitable direction.

In normal internal friction work, since the specimen diameter is very small, generally no error is introduced if it is assumed that the specimen temperature is continuously in phase with the temperature of the air in the furnace as the air temperature varies. When the specimen diameter is not small, this assumption is no longer valid, and the specimen temperature almost always lags behind the furnace air temperature under unsteady heating conditions. Ideally in this work, what is needed is a situation where the specimen temperature is raised

to the desired maximum temperature in as short a time as possible and to have the temperature constant while the internal friction measurement is being made, but reduce it as quickly as possible to the next lower temperature. An added complication is that it is not possible to attach any thermocouples directly onto the test specimen while the internal friction measurements are being made, so the measurement of the specimen temperature has to be indirect. This has been finally achieved by putting three thermocouples on the test specimen for calibration purposes and calibrating the central thermocouple against a fourth thermocouple measuring the air temperature in the furnace at a distance of about one-fourth of an inch from the central calibration thermocouple on the test specimen. The upper and bottom calibration thermocouples on the test specimen were used to establish the settings of the various secondary heating elements for the different desired temperature levels. The speed of the blower fan for air circulation was determined by trial and error. By this process, after a number of trials, it was found possible to raise the specimen temperature to 200°C in about 12 minutes. The total period of measurement was about one hour and forty-five minutes. The calibration was repeated a number of times and the results were found to agree within $\pm 1^{\circ}\text{C}$. The first measurement which needed some adjustments of the reflected light image in front of the photocell slit took about 3 to 4 more minutes. The subsequent measurements during the decrease of the temperature in 5°C intervals took about $2\frac{1}{2}$ to 3 minutes. Generally about 30 minutes elapsed before the temperature dropped from 200°C to 150°C . Intervals of 10°C were used below 150°C and the measurements were discontinued as soon as the temperature went below 80°C .

The measurement of internal friction was made less susceptible to human error by measuring the number of cycles for decay through the width of a slit 0.025 inches set at a distance of 85 inches in front of the test specimen. This was accomplished by making the image of a thin lighted slit of about 0.005 inches coincide with a zero setting, mechanically deflect the inertia bar attached to the test specimen, and measure by an electronic counter the number of cycles it takes for the amplitude of free vibration to decrease through the width of the slit. The inner side of the slit was set at 6/20 inches from the zero position and width of the slit was 0.025 inches. Hence, if n is the number of cycles it takes for the amplitude to decay through the width of the slit, then the internal friction is given by

$$\delta = \frac{1}{n} \log_e \left(\frac{0.3 + 0.025}{0.3} \right) = \frac{800}{n} \times 10^{-4}$$

The frequency of the natural vibration was chosen so that the internal friction peak occurred at 185°C. This frequency was found to be 1.75 c.p.s. The maximum shear stress corresponding to the initial amplitude was 100 psi.

TEST SPECIMENS

The test specimens were machined from the aluminum 4 weight per cent copper stock supplied by the Aluminum Corporation of America in the form of 1/2 inch diameter rods. The test length of the specimen was 3 inches and the diameter was 1/8 inch. The specimens were solution treated in an oven at 550°C for 7 days and quenched in

cold water. In order to prevent any creep sag, the specimens were horizontally supported during the solution treatment in a suitable mount, and the whole mount with the specimen was quenched. In order to assure the quick heating of the test specimen in the testing machine furnace, the specimen surface was subsequently coated with a thin brushed layer of aquadag suspension. This reduced the time to heat the specimen to the maximum temperature by about 4 minutes. It was not found to affect the internal friction measurements in any manner, and, since the solubility of carbon in aluminum is insignificant even at much higher temperatures, it does not affect the present problem.

ACCURACY OF MEASUREMENTS

Internal friction measurements as low as 2×10^{-4} were made in this program of work, and it has been found possible to repeat measurements at room temperature within 3 per cent quite consistently. At elevated temperatures where the temperature was changing quite rapidly in this work, it was not found possible to check this accuracy. It may be mentioned that every once in a while a spurious result was obtained in the sense that it did not logically fit into the rest of the measurements. Such measurements were rejected, but these were rare. While it was found that the reproducibility at room temperature was excellent when once the test specimen was mounted, it was found that this was anything but the case if the specimen was removed from the testing machine and mounted again. Considerable care was found necessary to obtain reasonable reproducibility in the background damping

after the performance of each part of the test on a specimen, as will be explained in the following section. The testing in essence involved the measurement of the internal friction about 4 times, and each time it must be made certain that no extraneous damping was introduced due to loose screws, etc. If the background damping varied too much, the test specimen was rejected. These rejects were quite large. As was stated already, the internal friction at the peak varied by a substantial amount from specimen to specimen. The peak values ranged anywhere from 40×10^{-4} to 15×10^{-4} , the room temperature damping being 5×10^{-4} or less. This gave normalized peak values ranging from 35×10^{-4} to 10×10^{-4} . Ideally in this work, it was found that the larger the starting value of the initial peak, the easier it was to follow the decrease in the height of the peak without being confused by the background damping. Therefore, tests were performed on only those specimens which give an initial peak height of about 25×10^{-4} or higher. Berry and Nowick (Ref. 3) reported much higher initial peak values in their work for polycrystalline aluminum 4 per cent copper. Such high values were not observed in this work. When all these problems were taken into consideration, it turned out that for every one specimen that gave usable results, there were about 10 rejects.

Experimental Technique

With the above background it is possible to discuss the necessary experimental technique. The following are the essential steps involved.

1. All the specimens are to be solution treated for at least 7 days at a temperature above 1000° F to assure that all the copper is taken into solution and that there is no micro-segregation. There will be

a certain amount of grain growth during this period. From the standpoint of internal friction due to stress induced ordering this is distinctly advantageous as the grain boundary damping which contributes to the background will be decreased. However the large grains will also incidentally contribute to a smaller fatigue life as well as larger scatter in the results of fatigue life making it difficult to compare the results. The solution treating period of 7 days is an arbitrary compromise.

2. The internal friction δ of such a specimen is determined soon after quenching as a function of temperature about 4 times with one day intervals. This is to investigate stability of the initial peak and confirm the conclusions of Berry and Nowick (Ref. 3) over a longer period and find the limitations.

3. Determine the δ vs T for a solution treated and quenched specimen (T4 condition) and subject it to torsional fatigue at a chosen stress level for a suitable percentage of the estimated life. Subject the specimen after fatigue to a hold at 150°C for 1/2 hour and cool it to room temperature. Soon after, determine the δ vs T for the test specimen, repeat the fatigue stressing and the subsequent cycle about three more times. After about four times the results of the measurements may not be expected to be reliable.

4. Repeat the test indicated in (3) above for different stress levels to investigate the stress dependence of the precipitation process.

RESULTS

The tests have been performed for stress levels 10, 000, 12, 000, 14, 000 and 16, 000 psi. In several tests, the fatigue life in torsion at 16, 000 psi was too short to follow the precipitation process through. The results of the tests are presented in Figs. 1 through 5. Fig. 1 shows the variation in the height of the initial peak when determined four times with one day intervals. Fig. 2 shows the results obtained for fatigue stressing the specimen at 10, 000 psi and 160 c. p. s. The specimen was damaged at this stage and the test could not be carried to completion. Fig. 3 shows the results for fatigue stressing at 12, 000 psi. The specimen broke after 8 hours and 5 minutes at this stress level. The results for 14, 000 psi were similar and so in Figs. 4 and 5 only the peak heights were reported. The specimen at 14, 000 psi was damaged after 1 hour of stressing while being mounted back in the testing machine after tightening the screws. It has not been possible to perform any tests so far at 16, 000 psi. In Fig. 4, the peak heights are plotted against time of fatigue stressing and in Fig. 5, the percentage variations are given.

It appears from the results presented in Fig. 1 that the initial peak is fairly stable over a period of three days so that any changes in the height of the initial peak may be attributed to the effect of fatigue stressing. On this basis it appears that the reduction in heights of the peak observed in Fig. 2 and 3 may be attributed primarily to the effect of fatigue. It may be recalled that the reduction of height is due to the formation of G. P. II zones. It may be observed that the decrease in the peak height occurs at a faster rate in the initial stages of the fatigue

than in latter stages. The dependence of this decrease in peak height on stress amplitude is quite striking as shown in Fig. 5. As the stress level increased, the rate of decrease of the peak height in the beginning increased also. This appears to be a plausible type of stress dependence. Within the experimental period the second broader peak associated with the formation of the θ' phase was not noticed.

Discussion of Results

While the results are not completely conclusive, it appears reasonable to assume on the basis of the results presented here, that the accelerated precipitation of the hardening element from precipitation hardening alloys in T4 condition does in fact occur. The formation of the G. P. II zones observed here is a process that occurs a long time before the microscopic precipitation observed by Forsyth and Hanstock. In fact it may be said that this stage will not normally be observed by any optical means. Hence the technique described, while somewhat difficult experimentally, enables one to observe fatigue damage substantially ahead of any other experimental means of observation and it is amenable to quantitative interpretation since the height of the peak is directly proportional to the square of the free copper concentration. As we start with a known percentage of the solute, in each specimen it is possible to follow the accelerated precipitation process (and hence the generation of soft spots leading to fatigue damage) in a quantitative manner.

The results reported are only indicative of the trend and the feasibility of the method. Substantial additional work is needed on this material and preferably also aluminum 10 per cent magnesium alloy to explore the idea fully.

CONCLUSIONS

Internal friction techniques have been used to follow the precipitation of copper from aluminum 4 per cent copper in solution treated and quenched condition when subjected to torsional fatigue at different stress levels. It appears that enhanced precipitation takes place during the fatigue stressing. The result of this enhanced precipitation is conjectured to leave some regions depleted of the solute atoms thus developing soft spots where the fatigue damage is concentrated. The internal friction technique seems to offer, on the basis of the preliminary investigation, a quantitative means of following fatigue damage during a period where it is not observable by other experimental processes. Considerably more experimental work is needed to explore the idea thoroughly.

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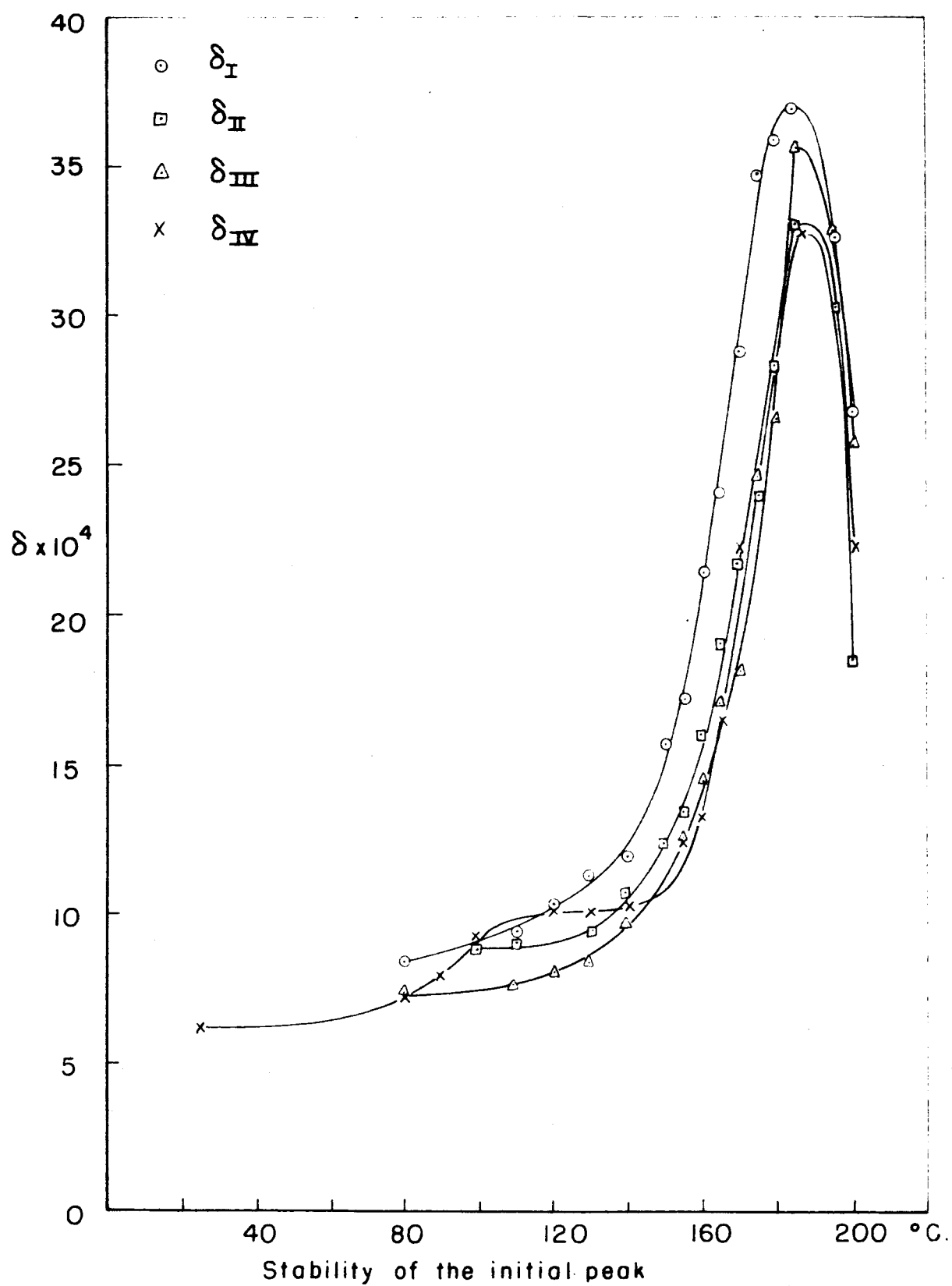


FIG. 1- REPEATED DETERMINATION OF THE INITIAL PEAK
OVER A PERIOD OF THREE DAYS

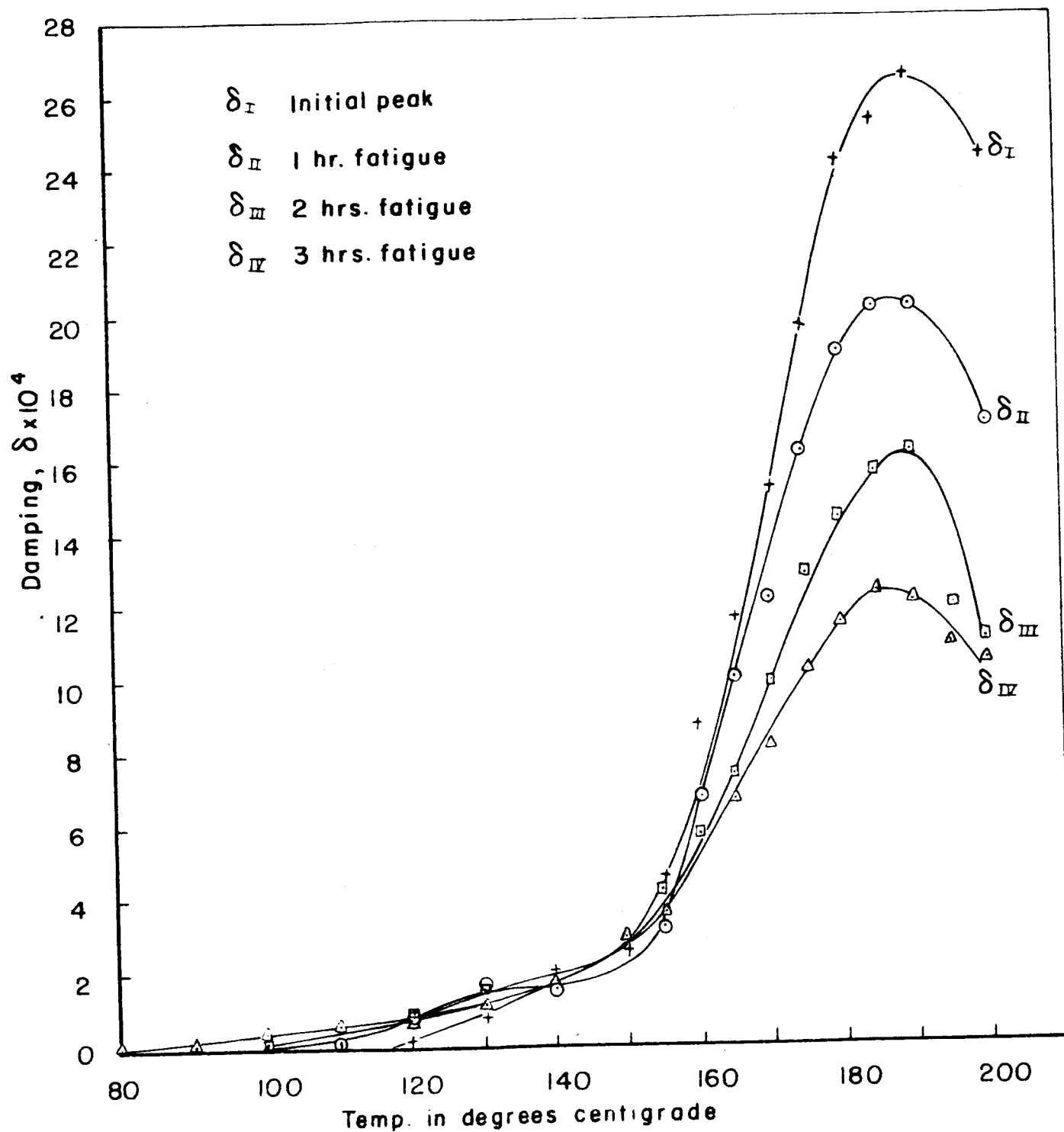


FIG. 2 - PRECIPITATION OF COPPER FROM AL. 4% CU. UNDER CONDITIONS OF TORSIONAL FATIGUE AT 10,000 PSI AND 160 C.P.S.

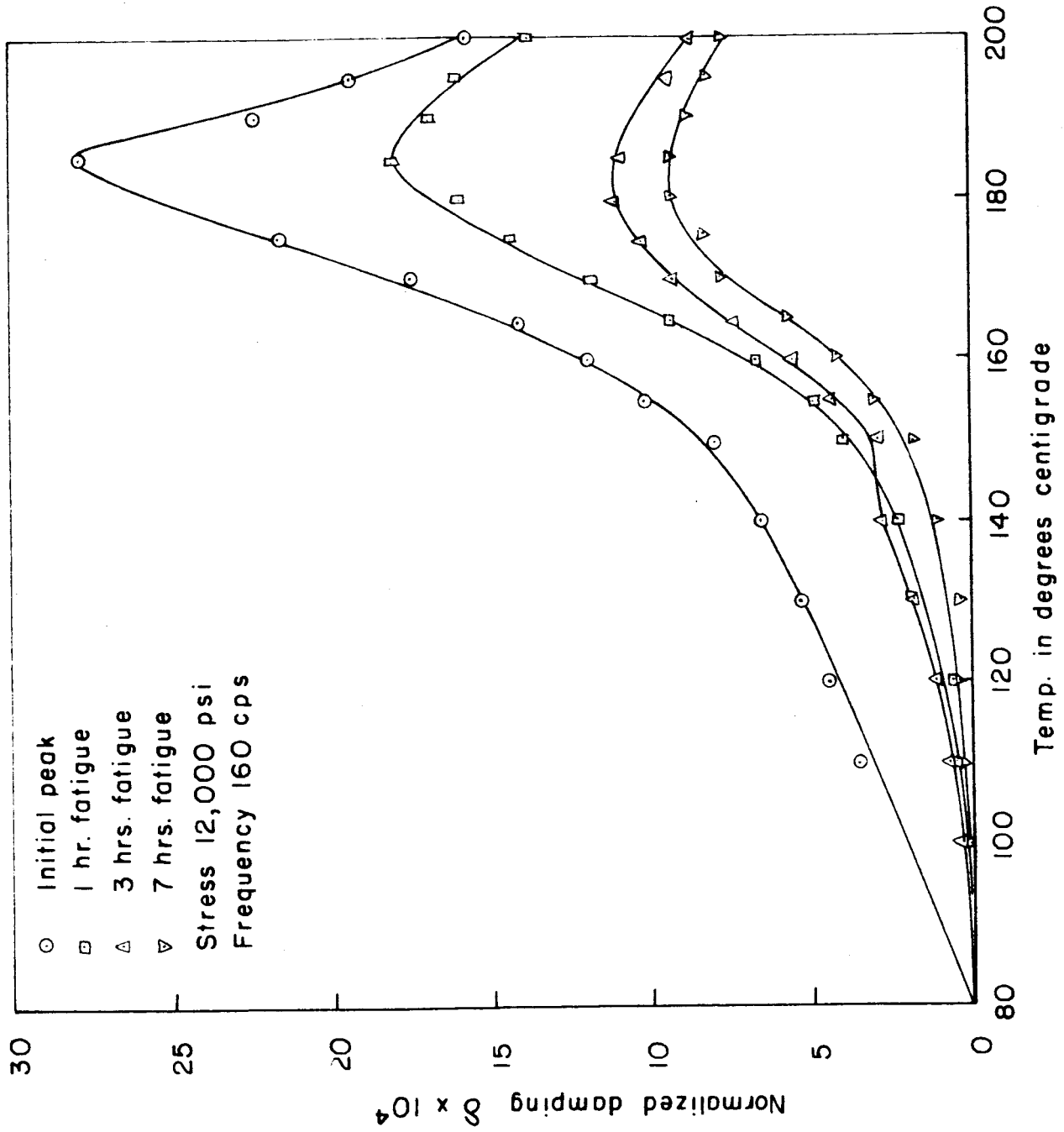


FIG. 3- PRECIPITATION OF COPPER FROM AL. 4% CU. UNDER CONDITIONS OF TORSIONAL FATIGUE AT 12,000 PSI AND 160 C.P.S.

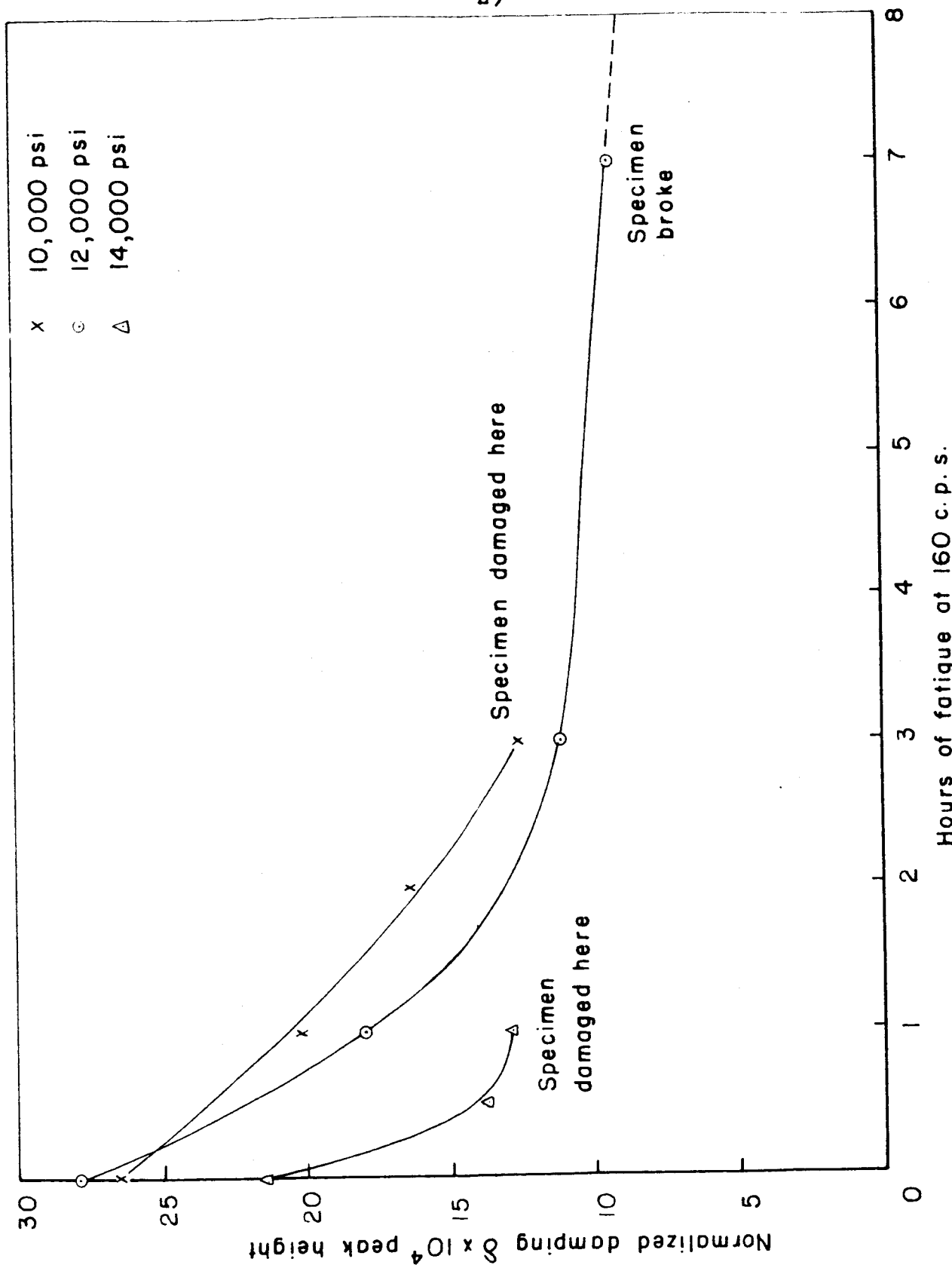


FIG. 4

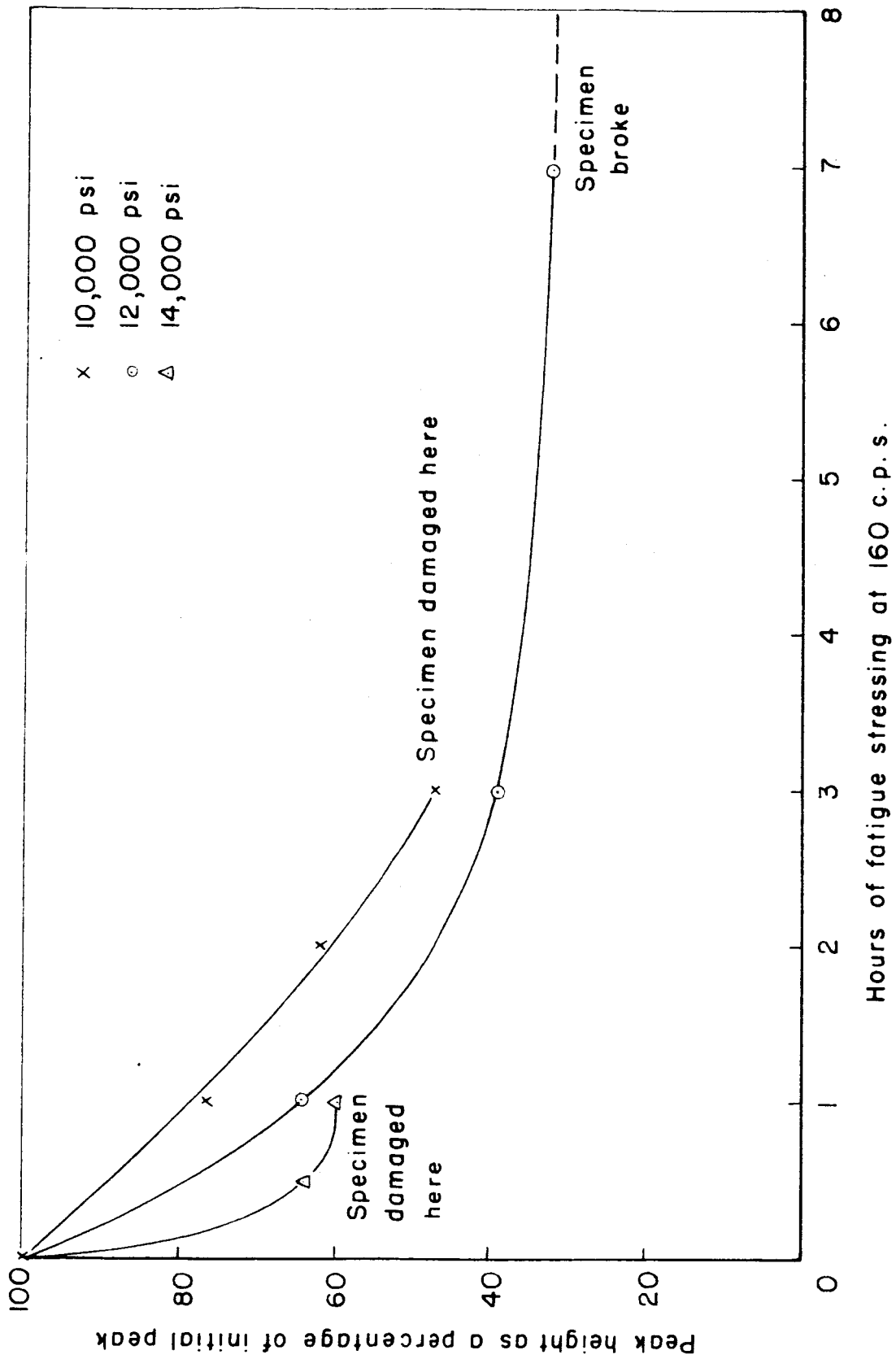


FIG. 5